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**PROVISIONAL PATENT APPLICATION**

ATTORNEY DOCKET NO.: 117196

DATE: September 16, 2003

**MAIL STOP PROVISIONAL PATENT APPLICATION**

**PROVISIONAL PATENT APPLICATION  
RULE §1.53(c)**

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Sir:

Transmitted herewith for filing under 37 CFR §1.53(c) is the provisional patent application of

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FOR (TITLE): PROCESS FOR PREPARING PURIFIED FATTY ACIDS

- ☐ Formal drawings (Figs. \_\_\_\_; sheets \_\_\_\_ ) are attached.  
☒ This patent application is assigned to AKZO NOBEL N.V.  
☐ The executed Assignment is filed herewith.  
☐ Entitlement to small entity status is hereby asserted.  
☒ Check No. 146379 in the amount of ☐ \$80.00 ☒ \$160.00 to cover the filing fee is attached.  
Except as otherwise noted herein, the Commissioner is hereby authorized to charge payment of any additional filing fees required under 37 C.F.R. §1.16 or credit any overpayment to Deposit Account No. 15-0461. Two duplicate copies of this sheet are attached.

Respectfully submitted,



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ATTORNEYS AT LAW

**Application Data Sheet****Application Information**

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CD-ROM or CD-R:	None
Title::	PROCESS FOR PREPARING PURIFIED FATTY ACIDS
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## PROCESS FOR PREPARING PURIFIED FATTY ACIDS

The present invention relates to a process for preparing free fatty acids derived  
5 from oils or fats in good yield and with good colour quality and colour stability,  
which process comprises (A) a hydrolytic splitting step of oils and/or fats to give  
a composition comprising crude fatty acids, (B) at least one thermal pre-  
treatment step of the composition obtained by step (A) to produce a thermally  
10 pre-treated composition, and (C) a high vacuum distillation step of the heat-  
treated composition obtained by step (B).

In the production process for the preparation of fatty acids, most often  
inexpensive starting materials are used, such as, natural oils or fats, or other  
fatty materials. The oils or fats are subjected to appropriate splitting conditions  
15 to release free fatty acids. Examples of such splitting processes include thermal  
splitting and enzymatic splitting. However, after the splitting step, the free fatty  
acids typically contain impurities, such as, unsplit or incompletely split  
glycerides, colour bodies, and compounds like sterols and phosphatides. In this  
respect, is noted that in this description the terms "crude fatty acids" and "crude  
20 fatty acid product" refer to free fatty acids that are contaminated with one or  
more of the above-mentioned impurities. Furthermore, the terms "free fatty  
acids" and "fatty acids" relate to aliphatic monocarboxylic acids having varying  
degree of unsaturation that are derived from glycerides (the esters of glycerol  
with said aliphatic monocarboxylic acids), said glycerides are widely available in  
25 nature as oils and fats. Furthermore, during the splitting process also  
undesirable polymerisation and decomposition reactions may take place, which  
results in additional contamination of the crude fatty acids. Another splitting  
process suitable to obtain free fatty acids from oil and fats is the alkali  
hydrolysis, commonly known as saponification. However, this splitting process  
30 has several drawbacks. One drawback is that this process requires a  
considerable amount of chemicals. More specifically, base (alkali) such as  
sodium or potassium hydroxide is used to do a full saponification of crude oils

and fats in order to obtain a fatty acid salt (soap). Afterwards, acid such as sulphuric or hydrochloric acid is needed to neutralise the reaction mixture and release the free fatty acids. The use of this base and acid makes the process economically and environmentally less viable, since an aqueous waste stream with large amounts of salts is produced during the process. A second drawback is that the saponification of crude oils and fats is often done as a batch process. Again, from an economical point of view this is not desirable.

A typical thermal or enzymatic splitting step provides two phases, i.e. an aqueous phase, which is a waste stream comprising glycerol, and a crude fatty acid product phase comprising about 95-99% by weight (wt.%) of free fatty acids, based on the weight of the crude fatty acid product and about 1-5 wt.% of impurities, such as the above-mentioned unsplit glycerides, colour bodies, sterols, phosphatides, but also minor amounts of water and other compounds may be present.

For the purification of crude fatty acids, several methods have been developed over the years. Distillation and/or fractionation are presently the most important of these (thermal) purification methods. Nowadays, many different distillation and/or fractionation processes are available, each process describing specific reaction conditions with respect to, for example, temperature and pressure, and each process specifically fine-tuned to provide pure fatty acids in good yield and with specific properties, for example, good colour quality and/or good colour stability.

An example of such a purification method is disclosed in WO 97/24420, which describes a process wherein purified polyunsaturated fatty acids and fatty acid ester mixtures that are essentially free of cholesterol and other sterols, and phosphorous compounds are derived from naturally occurring lipid mixtures. In the process of WO 97/24420, crude lipid mixture is first hydrolysed by the

addition of base or acid, to give a crude fatty acid product that is contaminated with, inter alia, sterols. Unreacted phospholipids and hydrolysed phospholipids act as surfactants, which may interfere with the formation of distinct crude fatty acids and aqueous phases. Therefore, it is preferred to add a separating aid in the form of one or more alcohols which facilitate the separation of the two phases. Next, the crude fatty acid product with sterols is subjected to thermal treatment at a temperature of 150-250°C. After this thermal treatment, the crude fatty acid mixture is distilled at a temperature of 130-250°C and a pressure of 0.001-0.53 kPa, which affords purified fatty acids. For the industrial preparation of common fatty acids however, the process of WO 97/24420 is economically not feasible, since it only involves steps that are conducted in a batch-wise fashion. Furthermore, the hydrolysis step in WO 97/24420, which is preferably a saponification step, requires the use of additional chemicals. As described before, this step produces an additional waste stream, and is also economically less viable because of the chemicals that are required.

US 2,862,943 describes a process for the purification of crude fatty acids to obtain fatty acids with improved colour. A small amount of boric acid compound is mixed with the crude fatty acids at the time of distillation. In some instances the crude fatty acid mixture is treated for a period of time at elevated temperatures of 100-300°C in the presence of the boric acid prior to the distillation. Thereafter, the crude fatty acid product is distilled at 0.667 kPa to give purified fatty acids. The process of US 2,862,943 requires the use of boric acid to achieve the objective and is therefore economically less profitable for industry.

EP 0,968,993 discloses a process for treating natural fatty acids before their subsequent distillation, wherein the fatty acids are heated to a temperature of 150-230°C and hydrogen peroxide is introduced in an amount of 0.5-5%, based on the amount of crude fatty acid to be treated. After completion of the



hydrogen peroxide introduction, a secondary reaction takes place at the same temperature for at least 10 minutes, the chromophores present in the crude fatty acids are converted to higher molecular weight substances, which remain in the residue in the subsequent distillation at 1 kPa. The use of hydrogen peroxide in the process of EP 0,968,993 makes the process economically less viable.

Finally, in US 2,583,028 a process is disclosed to improve the colour and colour stability of saturated higher fatty acids by treatment with a small quantity of  $\text{BF}_3$  at a temperature of 70-200°C for a period of 2-4 hours. Thereafter, the fatty acid products are distilled in vacuo in order to give a purified fatty acid product. The process of US 2,583,028 requires the use of  $\text{BF}_3$ , which is making the process economically less profitable.

A problem that is still unresolved by the prior art processes is to provide an economically feasible process wherein free fatty acids with good colour quality and colour stability are prepared in good yield. It is noted that the term "colour quality" refers to the colour just after distillation of the crude fatty acid product, whereas the term "colour stability" relates to the colour of the free fatty acids when measured after storage for a certain period of time at a specific temperature.

Hence, it is an objective of the present invention to provide a process overcoming this problem, said process being pre-eminently suited for preparing purified fatty acids in an economically attractive way and to produce free fatty acids with the above mentioned properties in good yield. We have now found that such free fatty acids can be obtained if the process comprises (A) a hydrolytic splitting step of oils and/or fats to give a composition comprising crude fatty acids and monoglycerides, (B) at least a thermal pre-treatment step of the composition obtained by step (A), and (C) distillation in a high vacuum

distillation unit of pre-treated composition obtained by step (B), wherein the composition that is obtained in the thermal pre-treatment step comprises a lower amount of monoglycerides compared to the amount of monoglycerides in the composition obtained by step (A) and is preferably below 0.5% by weight (wt.%) of monoglycerides, more preferably below 0.25 wt.% of monoglycerides, even more preferably below 0.1 wt.% of monoglycerides, and most preferably below 0.05 wt.% of monoglycerides, based on the weight of thermally pre-treated composition, with the proviso that the hydrolytic splitting step (A) is not a saponification step, because of the drawbacks mentioned before. Preferably, the product obtained by step (C) comprises below 0.5% by weight (wt.%) of monoglycerides, based on the weight of product after distillation and has a colour quality of Lr (5¼) below 0.6, preferably below 0.4 and Ly (5¼) below 5.5, preferably below 4.0, and a colour stability of Lr (5¼) below 1.9, preferably below 1.85 and Ly (5¼) below 24, preferably below 22.

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Accordingly, we claim a process for preparing a purified fatty acid with good colour quality and colour stability, said process comprising at least the steps of:

- (A) hydrolytic splitting of oils and/or fats to give a composition comprising crude fatty acids and monoglycerides,
- 20 (B) heat-treating the composition obtained by step (A) in a thermal pre-treatment unit, and
- (C) distilling the heat-treated composition obtained by step (B) in a high vacuum distillation unit,

wherein the composition that is obtained by the thermal pre-treatment step (B) comprises a lower amount of monoglycerides compared to the amount of monoglycerides in the composition obtained by step (A) and is preferably below 0.5% by weight (wt.%) of monoglycerides, more preferably below 0.25 wt.% of monoglycerides, even more preferably below 0.1 wt.% of monoglycerides, and most preferably below 0.05 wt.% of monoglycerides, based on the weight of thermally pre-treated composition, with the proviso that the hydrolytic splitting

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step (A) is not a saponification step. Typically, the composition that is obtained by the thermal pre-treatment step (B) comprises at least 0.00001 wt.% of monoglycerides, based on the weight of thermally pre-treated composition.

5

Processes according to the present invention may be a batch, semi-batch, or continuous processes. From an economic point of view, it is preferred to have continuous processes.

- 10 In a preferred embodiment of the present invention at least part of the residue that is obtained by the distillation step (C) is recycled to step (A).

- In another preferred embodiment of the present invention, the composition that is obtained by the thermal pre-treatment step (B) comprises at least 80% by weight (wt.%) of free fatty acids, preferably at least 85 wt.% of free fatty acids, more preferably at least 90 wt.% of free fatty acids, and most preferably at least 93 wt.% of free fatty acids, based on the weight of the thermally pre-treated composition. Typically, the composition that is obtained by the thermal pre-treatment step (B) comprises at most 99.99 wt.% of free fatty acids, based on the weight of the thermally pre-treated composition
- 15
- 20

- Suitable oils and fats that are preferably used in step (A) of the present process are selected from the group comprising of crude or refined vegetable oils and fats, crude or refined animal fats and oils, and acid oils. More preferred is the use of crude or refined vegetable oils and fats, and most preferred is the use of crude or refined vegetable oils and fats that, after hydrolytic splitting of said oils and fats, provide saturated or unsaturated, optionally hydroxy containing, crude fatty acids with 6 to 24 carbon atoms, or mixtures thereof. Examples of particularly preferred vegetable oils and fats include coconut oil, palm oil, palmkernel oil, sunflower oil, soybean oil, rapeseed oil, high erucic rapeseed oil,
- 25
- 30

castor oil, linseed oil, safflower oil, corn oil, cottonseed oil, groundnut oil, and canola oil.

Hydrolytic splitting processes that are suitable to release crude fatty acids from the oils and fats are conventional splitting processes, such as, thermal high pressure splitting and enzymatic splitting, which are extensively described in the art and are well-known to a man skilled in the art. Preferably, no separating aids, such as, alcohols are used to facilitate the formation of distinct crude fatty acids and aqueous phases. For economic and environmental reasons as explained above in more detail, the use of saponification as hydrolytic splitting technique is excluded from this invention.

It is noted that for commercial products that are produced on large scale, it is important that these products can be prepared in a cost efficient way and that the quality properties are suitable for the various industrial applications. For fatty acids, colour (quality) and colour stability are important quality properties. As described above, crude fatty acids derived by hydrolytic splitting of oils and fats often contain impurities like unsplit and incompletely-split glycerides, substances like sterols and phosphatides, colour bodies, and polymerisation and decomposition products. Colour quality and colour stability of fatty acids are influenced by the presence of, inter alia, monoglycerides and other colour bodies. An additional drawback of the presence of monoglycerides is that these compounds may harm the application performance of the final fatty acid product. Consequently, it is highly preferred to produce purified fatty acids that are essentially monoglyceride free. With the term "essentially free" is meant here that the amount of monoglycerides is such that any remaining amount of monoglyceride does not harm the colour quality and colour stability of the fatty acid product, nor the application performance of the final fatty acid product. The final fatty acid product, i.e. free fatty acids that are obtained after distillation step (C), preferably comprises below 0.5% by weight (wt.%) of monoglycerides,

more preferably below 0.25 wt.%, even more preferably below 0.1 wt.% of monoglycerides, and most preferably below 0.05 wt.%, based on the weight of the final fatty acid product. Typically, the final fatty acid product that is obtained after the distillation step (C) comprises at least 0.00001 wt.% of monoglycerides, based on the weight of the final fatty acid product.

During the thermal pre-treatment step (B), several reactions may take place. According to the present invention, monoglycerides that are present in the composition obtained by step (A) are reesterified under the thermal pre-treatment conditions to form higher boiling compounds, mainly di- and triglycerides. These di- and triglycerides remain in the residue during the subsequent distillation step (C). Because of the reesterification of monoglycerides, the crude fatty acid-containing composition that is obtained after the thermal pre-treatment step comprises a lower amount of monoglycerides compared to the amount of monoglycerides in the composition obtained by step (A). The thermal pre-treatment step (B) of the present invention provides a thermally pre-treated composition comprising an amount of preferably below 0.5% by weight (wt.%) of monoglycerides, more preferably below 0.25 wt.% of monoglycerides, even more preferably below 0.1 wt.% of monoglycerides, and most preferably below 0.05 wt.% of monoglycerides, based on the weight of thermally pre-treated composition. Furthermore, in addition to a reduced amount of monoglycerides obtained by step (B), it is advantageous if the thermally pre-treated composition comprises a high amount of free fatty acids. Particularly advantageous is a thermally pre-treated composition comprising at least 80% by weight (wt.%) of free fatty acids, preferably at least 85 wt.% of free fatty acids, more preferably at least 90 wt.% of free fatty acids, and most preferably at least 93 wt.% of free fatty acids, based on the weight of the thermally pre-treated composition. Since monoglycerides have a substantial influence on the performance, as well as the colour quality and colour stability of the final fatty acid product, the

reesterification of these monoglycerides during the thermal pre-treatment step is a very effective and desirable reaction. Additional reactions that may also take place during the thermal pre-treatment step are the polymerisation and/or decomposition reactions of (reactive) coloured bodies. Decomposed coloured  
5 bodies can be collected as separate fractions during the subsequent distillation step, whereas polymerised coloured bodies remain in the residue after distillation. The present process comprises at least one thermal pre-treatment step, although it also possible to carry out this thermal pre-treatment step more than once. It is preferred to do just one thermal pre-treatment step.

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The thermal treatment step (B) can be conducted in a thermal pre-treatment unit that is working in a batch, semi-batch, or continuous fashion. The thermal pre-treatment step may be conducted at a temperature in the range of 150-280°C, more preferably at a temperature of 200-250°C, and most preferably at  
15 a temperature of about 225°C. If the residence time of the crude fatty acids in the thermal pre-treatment unit is too long, undesirable reactions may occur which disadvantageously affect the overall yield of the present process. On the other hand, if the residence time of the crude fatty acid in the thermal pre-treatment unit is too short, impurities may remain in the final fatty acid product  
20 that can have a detrimental influence on the colour quality and colour stability of the final fatty acid product.

For economic feasibility of the overall process, it is particularly preferred that the thermal pre-treatment unit in step (B) is a continuously working unit. The  
25 reaction conditions in this thermal pre-treatment unit are such that the composition obtained by step (A) that is going through this continuously working unit has a narrow residence time distribution. The residence time distribution  $\phi$  is a dimensionless number that for a conventional stirred tank reactor generally varies between 0.2 and 6.0 ( $\phi = t / t'$ , wherein  $t$  is the actual residence time of a  
30 component (of a composition) and  $t'$  is the average residence time of all

components (of a composition)). Typically, for a narrow residence time distribution,  $\phi$  is at least 0.7-1.5 and in the most preferred situation each of these numbers approximate to 1. It is noted that only in the case of a batch process the residence time distribution  $\phi$  is 1. For batch, semi-batch, and continuous thermal pre-treatments applies that the average residence time associated with the temperature at which the thermal pre-treatment is preferably carried out varies from approximately 10 minutes to 6 hours. At a temperature of 150°C the average residence time is approximately 6 hours and at a temperature of 280°C the average residence time is approximately 10 minutes. At the most preferred temperature of about 225°C, the average residence time is approximately 1 hour.

Preferably, the process of the present invention does not include a washing and subsequent drying step between the thermal pre-treatment step (B) and the distillation step (C).

The high vacuum distillation step (C) of the present invention is based on a high vacuum distillation method, for example, thin film evaporation, (centrifugal) molecular distillation, wiped film distillation, or falling film distillation. Each of these distillation technologies allows the use of very low pressure (high vacuum) during the distillation process of 0.0001-0.2 kPa. For the present process, preferably a pressure of below 0.01 kPa and a temperature of 100-190°C are applied for the distillation of the thermally pre-treated composition. More preferably, a pressure of 0.001 kPa or less and a temperature of 150°C or less are applied. Preferably, the high vacuum distillation step (C) is based on thin film evaporation. The principle of thin film evaporation in the process of the present invention is that thermally pre-treated composition is heated in a device that is suitable to form a thin film of the composition material, which can be achieved, for example, by rotating the device or by applying a rotating wiper system. The formation of a thin film allows a gentle and steady evaporation of

- the various components that are present in the thermally pre-treated composition. Because a thin film allows quick evaporation, the residence time of the components of the composition in the heated device is short. This short residence time prevents the occurrence of undesirable polymerisation and decomposition reactions. A particularly preferred thin film distillation method is short path distillation. In a short path distillation unit, the distance between the evaporation area and the condensing area is much shorter compared to conventional distillation units.
- 5
- 10 In one embodiment of the present invention, at least part of the residue of the distillation step (C) is recirculated to step (A). Residue that is recirculated to step (A) is either admixed with newly added oils and/or fats prior to or during the hydrolytic splitting process or collected separately and recycled without admixing with newly added oils and/or fats. Although the residue may be
- 15 continuously recirculated, it will be obvious that after some recycle loops the residue is contaminated with undesired components to such an extent that it is more desirable to discard the residue. Alternatively, part of the recycle may be bled off to get a constant level of contaminants in the process.
- 20 Optionally, one or more conventional additives that are advantageous for the removal of coloured bodies may be used during the present process. Examples of these additives include, but are not limited to, phosphoric acid, hydrogen peroxide, sulphuric acid, boric acid, and mixtures thereof. These additives may be added to and admixed with the fatty acid containing composition in varying
- 25 concentrations after step (A) and before, during, or after the thermal pre-treatment step (B), but in any case prior to the high vacuum distillation step (C). The use of one or more additives during distillation step (C) is undesired since small amounts of one or more of these additives may end up in the final product. Preferably, conventional additives are essentially not used, wherein
- 30 the term "essentially not used" relates to amounts that are below conventionally



used amounts. For example, in US 2,862,942 this conventional amount is 0.001-1.0% of boric acid, in US 2,583,028 this conventional amount is 0.25-1% of boron trifluoride (BF<sub>3</sub>), and in EP 0,968,993 this conventional amount is 0.5-5% of hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), all based on the amount of crude fatty acid to be treated.

The presence of mono-, di-, and/or triglycerides and possible other components in a composition comprising free fatty acids may be determined by conventional Gas Chromatography (GC) methods, or Gel Permeation Chromatography (GPC) techniques, prior, during, or after any of the steps of the present process. GPC is a high performance liquid chromatography technique for the separation of components based on their molecular size in solution. GPC separates the sample into its discrete components and determines the molecular weight distribution of a sample.

15

In order to further elucidate the present invention, the following examples are given as an illustration:

#### High Pressure Splitting (HPS)

20 About 200 tons (t) of crude linseed oil is split in a commercial continuous countercurrent splitter at 230°C and 33 bar (with a feed rate of 4.5 t/h). The oil is introduced from the bottom, water from the top. Difference in densities combined with the pumping force with which crude linseed oil is introduced to the countercurrent splitter causes mixing of the oily and aqueous currents. The

25 crude fatty acids (i.e. split oil product comprising free fatty acids, mono-, di-, and triglycerides) that are obtained are isolated from the aqueous waste stream comprising glycerol, collected and analysed (Table 1). Optionally, part of the isolated and collected crude fatty acids may be recirculated to the countercurrent splitter and admixed with newly added crude linseed oil at the

30 bottom of the splitter, which will reduce the induction phase of the splitting

process.

Table 1:

Components analysed by GPC	Crude linseed oil (wt.%) <sup>1</sup>	High pressure splitting
		Crude fatty acids (wt.%) <sup>2</sup>
Monomer (FFA) <sup>3</sup> content	<3 <sup>4</sup>	89.4
Monoglyceride content		2.8
Diglyceride content		6.9
Triglyceride content	>97 <sup>4</sup>	0.9

- 1) Weight percent, based on the total amount of crude linseed oil.
- 5 2) Weight percent, based on the total amount of crude fatty acid after splitting.
- 3) FFA = Free fatty acid
- 4) Typical amount in a composition of naturally occurring crude linseed oil

#### Comparative Example A

- 10 The crude fatty acids obtained in the high pressure splitting are distilled in a conventional continuous distillation plant at 270°C and 0.5 kPa (with a feed rate of 4.3 t/h) as described in "Ed. R.W. Johnson, E. Fritz; Fatty Acids in Industry: Processes, properties, derivatives, applications; Marcel Dekker, Inc., New York, 1989". The yield is 83% of distilled product, based on the amount of
- 15 crude fatty acids that was distilled. Analysis of the residue indicated an amount of polymerised fatty acids of 10.3% as determined by GPC, based on the amount of residue. For analytical data see Table 2. About 50 t of residue that was obtained by this distillation was again subjected to the conditions of high pressure splitting as described above and subsequently the crude fatty acids
- 20 that were obtained by this splitting step were again subjected to the conventional continuous distillation conditions, to give a second amount of free fatty acids in about 50% yield.

Table 2:

Components analysed by GPC	Crude fatty acids by HPS (wt.%)	Comparative Example A	
		Product after dist. (wt.%) <sup>1</sup>	Product after 2nd dist. (wt.%) <sup>2</sup>
Monomer (FFA) content	89.4	~100	~100
Monoglyceride content	2.8	n.d. <sup>7</sup>	n.d. <sup>7</sup>
Diglyceride content	6.9	n.d. <sup>7</sup>	n.d. <sup>7</sup>
Triglyceride content	0.9	n.d. <sup>7</sup>	n.d. <sup>7</sup>
Colour quality <sup>3</sup>			
Lr (5%) <sup>5</sup>		0.8	0.8
Ly (5%) <sup>6</sup>		5.5	7.8
Colour stability <sup>4</sup>			
Lr (5%) <sup>5</sup>		1.9	-
Ly (5%) <sup>6</sup>		24	-

1) Weight percent, based on the total amount of product after distillation

2) Product after high pressure splitting of the residue of the first distillation and subsequent second distillation. Weight percent, based on the total amount of product after distillation

5 3) Fresh colour after distillation

4) Colour after 21 days storage at 45°C

5) Lr (5%) is Lovibond Red measured in a 5% inch cell

6) Ly (5%) is Lovibond Yellow measured in a 5% inch cell

7) Not detectable (n.d.), i.e. <0.05% of weight, based on the amount of product after distillation

10

#### Comparative Examples B and C

The crude fatty acids obtained by the high pressure splitting are distilled in a short path distillation unit at 150°C (Comparative Example B) or 175°C (Comparative Example C) and 0.002 kPa (with a feed of 400 kg/h) in both

15 Comparative Examples. The yields are 81% and 88% of distilled product,

based on the amount of crude fatty acids that was distilled, for Comparative Example B and Comparative Example C, respectively. For the analytical data see table 3.

5 Table 3:

Components analyzed by GPC	Crude fatty acids by HPS (wt.%)	Comp. Example B Product after dist. with SPD <sup>1</sup> at 150°C (wt.%) <sup>2</sup>	Comp. Example C Product after dist. with SPD <sup>1</sup> at 175°C (wt.%) <sup>2</sup>
Monomer (FFA) content	89.4	98.5	97.5
Monoglyceride content	2.8	1.5	2.5
Diglyceride content	6.9	n.d. <sup>7</sup>	n.d. <sup>7</sup>
Triglyceride content	0.9	n.d. <sup>7</sup>	n.d. <sup>7</sup>
<b>Colour quality<sup>3</sup></b>			
Lr (5¼) <sup>6</sup>		0.5	0.9
Ly (5¼) <sup>6</sup>		4.5	7.2
<b>Colour stability<sup>4</sup></b>			
Lr (5¼) <sup>6</sup>		3.2	5.2
Ly (5¼) <sup>6</sup>		36	52

1) SPD = Short Path Distillation

2) Weight percent, based on the total amount of product after distillation.

3) Fresh colour after distillation

4) Colour after 21 days storage at 45°C

10 5) Lr (5¼) is Lovibond Red measured in a 5¼ inch cell

6) Ly (5¼) is Lovibond Yellow measured in a 5¼ inch cell

7) Not detectable (n.d.), i.e. <0.05% of weight, based on the amount of product after distillation

**Example 1**

The crude fatty acids obtained by the high pressure splitting were pre-treated in a batch reactor by reesterification of the glycerides in the crude fatty acids at 220°C for 1h at 2 kPa . Next, the pre-treated crude fatty acid product was

5 distilled in a short path distillation unit at 150°C at 0.001 kPa (with a feed rate of 15.5 kg/h). The yield of distilled product after batch thermal pre-treatment and short path distillation was 83%. Analysis of the residue of the distillation indicated an amount of polymerised fatty acids of 4.7% as determined by GPC, based on the amount of residue. For the analytical data see table 4.

10

Table 4:

Components analyzed by GPC	Example 1		
	Crude fatty acids by HPS (wt.%)	Product after pre-treatment of crude fatty acids (wt.%) <sup>1</sup>	Product after pre-treatment and dist. (wt.%) <sup>2</sup>
Monomer (FFA) content	89.4	84.0	100
Monoglyceride content	2.8	n.d. <sup>7</sup>	n.d. <sup>7</sup>
Diglyceride content	6.9	8.2	n.d. <sup>7</sup>
Triglyceride content	0.9	7.8	n.d. <sup>7</sup>
Colour quality <sup>3</sup>			
Lr (5%) <sup>5</sup>			0.1
Ly (5%) <sup>6</sup>			1.8
Colour stability <sup>4</sup>			
Lr (5%) <sup>5</sup>			0.7
Ly (5%) <sup>6</sup>			12

1) Weight percent, based on the amount of crude fatty acids after pre-treatment.

2) Weight percent, based on the amount of pre-treated crude fatty acids after distillation.

3) Fresh colour after distillation

15 4) Colour after 21 days storage at 45°C

5) Lr (5%) is Lovibond Red measured in a 5% inch cell

6) Ly (5%) is Lovibond Yellow measured in a 5% inch cell

7) Not detectable (n.d.), i.e. <0.05% of weight, based on the amount of product after distillation

**Example 2**

The crude fatty acids obtained by the high pressure splitting were continuously fed at a specific rate  $R_1$  (feed rate  $R_1$  of 0.2 L/h) into a stirred 2 L glass reactor filled with 1.2 L of crude fatty acids at 200 °C. At the same rate  $R_1$  as product was fed into the reactor, an amount of product was removed from the reactor and then cooled to 30°C in a heat exchanger. The rate  $R_1$  was such that an average residence time of 360 minutes in the reactor was achieved. The cooled product was collected, analysed, and subsequently distilled in a short path distillation unit at 150°C and 0.001 kPa (feed rate  $R_2$  of 0.6 kg/h). The yield of distilled product was 84%. Analysis of the residue of the distillation indicated an amount of polymerised fatty acids of 5.9% as determined by GPC, based on the amount of residue. The analytical data are presented in Table 5. The residue was subjected three times to high pressure splitting conditions in a batch autoclave at 220°C for 2h each using an oil/water ratio of 1:1. The total amount of crude fatty acids that were obtained after these three treatments were then thermally pre-treated at 200°C for 1h. Finally, distillation of the thermally pre-treated crude fatty acids as described in Example 2 afforded free fatty acids in about 70% yield.

Table 5:

Components analyzed by GPC	Crude fatty acids by HPS (wt.%)	Example 2	
		Product after pre-treatment of crude fatty acids (wt.%) <sup>1</sup>	Product after pre-treatment and dist. (wt.%) <sup>2</sup>
Monomer (FFA) content	88.4	85.8	100
Monoglyceride content	2.8	n.d. <sup>7</sup>	n.d. <sup>7</sup>
Diglyceride content	6.9	9.3	n.d. <sup>7</sup>
Triglyceride content	0.9	4.8	n.d. <sup>7</sup>
Colour quality <sup>3</sup>			
Lr (5¼) <sup>5</sup>			0.3
Ly (5¼) <sup>6</sup>			3.6
Colour stability <sup>4</sup>			
Lr (5¼) <sup>5</sup>			1.8
Ly (5¼) <sup>6</sup>			16

1) Weight percent, based on the amount of crude fatty acids after pre-treatment.

2) Weight percent, based on the amount of pre-treated crude fatty acids after distillation.

3) Fresh colour after distillation

5 4) Colour after 21 days storage at 45°C

5) Lr (5¼) is Lovibond Red measured in a 5¼ inch cell

6) Ly (5¼) is Lovibond Yellow measured in a 5¼ inch cell

7) Not detectable (n.d.), i.e. <0.05% of weight, based on the amount of product after distillation

10 From these results it is clear that the process of the present invention provides free fatty acids in good yield and with good colour quality and colour stability.

**Claims**

1. Process for preparing a purified fatty acid, said process comprising at least the steps of:
  - 5 (A) hydrolytic splitting of oils and/or fats to give a composition comprising crude fatty acids and monoglycerides,
  - (B) heat-treating the composition obtained by step (A) in a thermal pre-treatment unit, and
  - 10 (C) distilling the heat-treated composition obtained by step (B) in a high vacuum distillation unit,wherein the composition that is obtained by the thermal pre-treatment step (B) comprises a lower amount of monoglycerides compared to the amount of monoglycerides present in the composition obtained by step (A) and is preferably below 0.5% by weight (wt.%) of monoglycerides, more preferably  
15 below 0.25 wt.% of monoglycerides, even more preferably below 0.1 wt.% of monoglycerides, and most preferably below 0.05 wt.% of monoglycerides, based on the weight of thermally pre-treated composition, with the proviso that the hydrolytic splitting step (A) is not a saponification step.
- 20 2. Process according to claim 1 wherein the heat-treatment in step (B) is conducted at a temperature of 150-280°C, more preferably at a temperature of 200-250°C, and most preferably at a temperature of about 225°C.
- 25 3. Process according to claim 1 or 2 wherein in step (B) the composition is heat-treated in a continuously running thermal pre-treatment unit, preferably in a unit wherein the components of the composition have a residence time distribution of 0.7-1.5.
- 30 4. Process according to any one of claims 1-3 wherein the composition that is obtained by the thermal pre-treatment step (B) comprises at least 80% by



weight (wt.%) of free fatty acids, preferably at least 85 wt.% of free fatty acids, more preferably at least 90 wt.% of free fatty acids, and most preferably at least 93 wt.% of free fatty acids, based on the weight of the thermally pre-treated composition.

5

5. Process according to any one of claims 1-4 wherein in step (C) the heat-treated composition of step (B) is distilled at a pressure of below 0.01 kPa and at a temperature of 100 to 190°C, preferably at a pressure of 0.001 kPa or less and a temperature of 150°C or less.

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6. Process according to any one claims 1-5 wherein step (C) is conducted in a short path distillation unit.

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7. Process according to any one claims 1-6 wherein at least part of the residue that is obtained by the distillation step (C) is recycled to step (A).

20

8. Process according to any one of claims 1-7, wherein the oils and/or fats that are subjected to the hydrolytic splitting in step (A) are selected from the group consisting of crude or refined vegetable oils and fats, crude or refined animal oils and fats, and acid oils, preferably selected from crude or refined vegetable oils and fats, most preferably from crude or refined vegetable oils and fats that, after hydrolytic splitting of said oils and fats, provide saturated or unsaturated, optionally hydroxy containing, crude fatty acids with 6 to 24 carbon atoms, or mixtures thereof.

25

9. Process according to any one of claims 1-8, wherein one or more conventional additives are used before, during, or after the thermal pre-treatment step (B), but in any case after the hydrolytic splitting step (A) and prior to the distillation step (C), said additives being effective for the removal of colour bodies.

30

**Abstract**

The present invention relates to a process for preparing fatty acids derived from oils and fats in good yield and with good colour quality and colour stability, which process comprises the steps of (A) hydrolytic splitting of oils and/or fats, (B) at least one thermal pre-treatment of a composition comprising crude fatty acids and monoglycerides, and (C) high vacuum distillation of thermally pre-treated composition comprising crude fatty acids.

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